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by
R. E. Taylor and D. P. DeWitt

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School of Mechanical Engineering

Purdue University, West Lafayette, Indiana

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1. Research Objectives

The overall research objective was to develop a state-of-the-art emissometer capable of measuring spectral normal emissivity of samples of electrically conducting materials, of non-conducting opaque materials and to demonstrate the feasibility of measuring semi-transparent materials at high temperatures. The successful conclusion of this objective has resulted in a unique apparatus that has been used in high technological applications such as laser hardening materials, carbon/carbon composites, turbine blades, combustor liners and electronic materials processing. The apparatus has been developed to the point where operation on opaque materials, both electrical conductors and insulators, has become a routine operation. The various parameters that control the accuracy of the measurements, such as black body quality, temperature gradients and veiling glare effect have been studied extensively. Test results have been obtained on tantalum, silicon carbide, silicon nitride, carbon/carbon composites, pyroceram, and oxidized Rene at temperatures between 1300 and 3000 K which have demonstrated the accuracy and reproducibility of the apparatus above 1300 K. The present work has developed the basis for measuring semi-transparent materials.

2. Status of the Research

A. Introduction

The spectral emissivity of materials is an important property from the standpoint of heat transfer, surface studies including oxidation and thin films, detection and analysis (signatures), and laser-related technology including vulnerability. High technology applications such as turbine blades, combustor liners, laser hardening systems, and electronic materials processing have stringent requirements for radiative properties of advanced materials, particularly nonconducting ones, at high temperature approaching their destruction/degradation temperatures in the infrared spectral region. Unfortunately, few laboratories have the capability to provide these property measurements. Therefore it is important that the capability for determining this property to a high degree of accuracy be readily available to the nation.

The Multiproperty Apparatus is a unique and extremely important device that was developed at the Thermophysical Properties Research Laboratory at Purdue University. It has proven to be a powerful scientific and engineering tool for experimentally determining several thermophysical properties at high temperatures. The emissometer was developed to extend the capabilities of the Multiproperty Apparatus to include measurements of the normal spectral emissivity of conducting and nonconducting materials. Previously the emissometer was developed to the point where state-of-the-art accuracy from 2 to 15 μm was obtained on opaque materials above 1300 K. Measurements were made on several materials. In the case of electrical conductors, results were obtained on tantalum (used as a standard) and on a carbon/carbon composite. In the case of non-conductors, measurements were made on a number of silicon carbides and silicon nitrides fabricated at eight facilities using a variety of techniques. Also preliminary measurements were made on pyroceram, a material of interest in certain defense applications. The present year's effort included finishing the work on pyroceram, reducing the measurement for metallic materials to a routine operation and demonstrating this by measuring the properties of certain platinum alloys and oxidized Rene, extending the lower wavelength limit from 2 down to 0.4 μm , and by performing a feasibility analysis for extending the capabilities to measure semi-transparent materials.

B. Emissometer Improvements

Several improvements were made in the emissometer in order to improve the ease of operation, extend its capabilities and to increase the accuracy obtainable. One of the major problems associated with the operation of the emissometer has been the problem of realignment each time the emissometer table is positioned in front of the Multiproperty Apparatus. During many applications involving the Multiproperty Apparatus, the area in front of the apparatus is used for pyrometry and for twin-telemicroscopes. However, when spectral emissivity measurements are to be performed the pyrometer and telemicroscopes are removed and the emissometer table is positioned in front of the apparatus. Then an extensive optical alignment had to be performed. However, this procedure has been greatly simplified and now it is possible to move the table into position and align the emissometer within an hour.

Measurements of spectral emissivity were limited to the region 2 to 15 μm by the detectors previously used. During the past year we have purchased a relatively inexpensive silicon detector which permits measurements in the 0.4 to 1.5 μm range and a high quality liquid-nitrogen-cooled trimetal detector for use in the 1 to 5 μm range. We have demonstrated the usefulness of these detectors in addition to our standard detector and we now can make measurements from 0.6 to 15 μm (see Figure).

As a result of further analysis of our integral-blackbody technique for opaque nonconducting solids it was discovered that the quality of our blackbody hole at longer wavelengths was not as high as we had estimated. This problem is caused by the very low emissivity at longer wavelengths for tantalum which serves as the heating tube. This problem can be overcome by inserting a graphite block in the region of the blackbody hole and by viewing the holes at least 15° off-axis or by using a material which has a much larger emissivity - for example, oxidized Rene.

Refinements were made in our finite-difference heat transfer analysis and temperature gradients existing in opaque non-electrical conductors were examined in detail. The results show that the gradients are acceptable and will not cause large errors in the emissivity results for most materials.

C. Measurement Results

Two samples of oxidized Rene were measured using the emissometer with three detectors and two prisms, as well as with two windows (NaCl and quartz) on the Multiproperty Apparatus. The results in the overlapping wavelength region were within 1%. In addition measurements were made at 0.65 μm using an automatic optical pyrometer and these results were within 1.5% of those obtained using the emissometer. The results are shown in the Figure. Measurements were also made on proprietary samples of platinum alloy.

The study on pyroceram was completed. The results showed a strong dependence on material history below 3 μm but that the temperature dependency up to the softening point above 5 μm was essentially zero. The data also revealed the existence of a "valley" of about 10% in the vicinity 10.6 μm , which is a wavelength region of high interest for laser applications.

A feasibility study on the measurement of semi-transparent results was performed. The results, including some experimental evidence, delineated the procedures required to measure this type of material and has involved two activities. The first activity has been the analysis of different sample-heating tube configurations for which models have been developed to relate the optical properties (n , index of refraction, k absorption coefficient, and ϵ , emissivity) to the experimental radiance observations. Two major conclusions have been reached from this analysis: (a) the simplest experimental arrangement which renders the temperature gradients negligible involves significant modifications to the Multi-Property Apparatus, and (b) a simplified experimental arrangement in which significant temperature gradients exist, but their effects estimated, is attractive. Toward this end we will upgrade our heat transfer analysis of the sample-heating tube to include semitransparent materials. The second activity has been the measurement of commercial-grade fused quartz using the opaque approach (lateral and blackbody holes) in addition to a through hole. Preliminary measurements have been made but are still being evaluated. We will report upon this work in a subsequent publication.

3. Cumulative Chronological List of Written Publications in Technical Journals

D.P. DeWitt, R.E. Taylor and T.K. Riddle, "High Temperature Computer Controlled Emissometer for Spectral and Total Measurements on Conducting and Nonconducting Materials," Proceedings Seventh Symposium on Thermophysical Properties, ASME, pp. 256-64, 1977.

R.E. Taylor and H. Groot, "Thermophysical Properties of POCO Graphite," High Temperature-High Pressure, 12, pp. 147-60, 1980.

P.E. Johnson, D.P. DeWitt and R.E. Taylor, "Method for Measuring High Temperature Spectral Emissivity of Nonconducting Materials," AIAA Journal, 19(1), 113-20, Jan. 1981.

D.P. DeWitt, R.E. Taylor, H.M. James, and A. Ono, "High Temperature Spectral Emissivity of Conducting and Nonconducting Materials," Proceedings Eighth Symposium on Thermophysical Properties, ASME, in preparation.

R.E. Taylor, "Thermophysical Property Determinations Using Direct Heating Methods," High Temperature-High Pressure, in press.

4. Technical Reports and Thesis prepared for this Project

- (1) D.P. DeWitt, B. Hoenle and R.E. Taylor, "Spectral and Total Emissivity and Reflectivity at High Temperature," April 30, 1976, PRL Report 116.
- (2) D.P. DeWitt, R.E. Taylor and B.D. Hoenle, "Description of the PRL High Temperature Emissometer," November 1976, PRL Report 126.
- (3) R.E. Taylor and D.P. DeWitt, "Spectral and Total Emissivity and Reflectivity at High Temperatures," March 31, 1977, PRL Report 130.
- (4) D.P. DeWitt, T.K. Riddle and R.E. Taylor, "Spectral and Total Emissivity and Reflectivity at High Temperatures," Profess Report to AFOSR, September 15, 1977, PRL Report 136.
- (5) D.P. DeWitt, T.K. Riddle and R.E. Taylor, "Spectral and Total Emissivity and Relectivity at High Temperatures," Interim Report to AFOSR, April 23, 1978, AFOSR-TR-78-0901, PRL Report 152.
- (6) R.E. Taylor and H. Groot, "Thermal Properties of POCO Graphite," May, 1978, AFOSR-TR-78-1375, PRL Report 153.
- (7) P.E. Johnson, D.P. DeWitt and R.E. Taylor, "Spectral Emissivity at High Temperatures, Six Month's Report to AFOSR," September 1978, AFOSR-TR-78-1480, PRL Report 163.
- (8) T.K. Riddle, "A New Method for Measuring the Spectral Emissivity of Nonconducting Materials at High Temperatures," May 1978, MSME Thesis, School of Mechanical Engineering, Purdue University.
- (9) R.E. Taylor, D.P. DeWitt and P.E. Johnson, "Spectral Emissivity at High Temperatures," Annual Report, April 1979, TPRL 189.
- (10) R.E. Taylor, D.P. DeWitt and P.E. Johnson, "Spectral Emissivity at High Temperatures, Annual Report, April 1980, TPRL 216.
- (11) P.E. Johnson, "High Temperature Normal Spectral Emissivity of Several Important Technical Materials," August 1980, MSME Thesis, School of Mechanical Engineering, Purdue University.
- (12) R.E. Taylor, "Specific Heat of Carbon/Carbon Composites," October 1980, AFOSR-TR-80-1360, TPRL Report 235.
- (13) R.E. Taylor and H. Groot, "Thermal Diffusivity of HEPN Rocket Nozzle," May 1980, TPRL 244.
- (14) D.P. DeWitt and R.E. Taylor, "Spectral Emissivity for High Temperature Measurements on Opaque Materials," in preparation.
- (15) D.P. DeWitt, R.E. Taylor, A. Ono, and H.M. James, "Spectral Emissometer for Semi-Transparent Materials," in preparation.

5. List of the Professional Personnel Associated with the Research Effort

D.P. DeWitt, Professor Mechanical Engineering, Co-major investigator, responsible for radiometric related procedures and instrumentation.

R.E. Taylor, Senior Researcher, responsible for multi-properties apparatus, digital data acquisition system and materials science aspects.

T.K. Riddle, Graduate Student, MSME May 1978 Thesis, "A New Method for Measuring the Spectral Emissivity of Nonconducting Materials at High Temperatures."

P.E. Johnson, Graduate Student, MSME August 1980 Thesis, "High Temperature Normal Spectral Emissivity of Several Important Technical Materials."

R.L. Shoemaker, Associate Researcher, assisted in computer-related problems.

H.M. James, Professor of Physics, assisted in theoretical analyses of radiometric considerations in semi-transparent materials.

A. Ono, Visiting Professor, National Laboratory for Metrology (Japan), assisted in radiometric procedures and instrumentation.

W. Vaughn, (Research Assistant) assisted in machining operations.

6. Interactions

A. Spoken papers presented at meetings

- (1) D.P. DeWitt, R.E. Taylor and T.K. Riddle, "High Temperature Computer Controlled Emissometer for Spectral and Total Measurements on Conducting and Nonconducting Materials," presented at the Seventh Symposium on Thermophysical Properties, ASME, Gaithersburg, MD, May 10-12, 1977.
- (2) P.E. Johnson, D.P. DeWitt and R.E. Taylor, "Method for Measuring High Temperature Spectral Emissivity of Nonconducting Materials," presented at the 14th AIAA Thermophysics Conference, June 4-6, 1979, Orlando, Florida.
- (3) D.P. DeWitt, P.E. Johnson and R.E. Taylor, "Spectral Emissivity of Ceramics at High Temperatures: Silicon Carbide and Silicon Nitride," paper 79-HT-24, 18th ASME-AIChE National Heat Transfer Conference, August 5-8, 1979, San Diego, CA.
- (4) R.E. Taylor, "Thermophysical Property Determinations Using Direct Electrical Heating," presented at the 7th European Thermophysical Properties Conference, June 1980, Antwerp.

- (5) D.P. DeWitt, R.E. Taylor, H.M. James, and A. Ono, "High Temperature Spectral Emissivity of Conducting and Nonconducting Materials," to be presented at the Eighth Symposium on Thermophysical Properties, ASME, Gaithersburg, MD, June 1981.
- (6) R.E. Taylor, "Thermal Diffusivity Measurements of HEPN," to be presented at the 3rd Carbon-Carbon Nozzle Technology Meeting to be held October, 14-15, 1981.

B. Consultive and Advisory Functions

- (1) Mr. Koenig of AFML has sponsored a small grant to measure the emissivity of pyroceram to the softening point. We have measured this material up through the softening region.
- (2) We have measured the total emissivity of certain graphitic materials and provided consulting services for the Department of Energy (C. Tarr) for the instrument power system of the Solar Polar Launch Vehicle.
- (3) We have measured the spectral emissivity of several platinum alloys for Owens Corning (K. Bubba).
- (4) We are providing consulting services for Oak Ridge National Laboratory (D. McElroy) in obtaining and installing a multiproperty apparatus for high temperature thermophysical property research.
- (5) We have measured the spectral properties of oxidized Rene for NASA.

7. Other Statements

- (a) Dr. A. Ono of the National Research Laboratory of Metrology of Japan has received a government grant to come to this country on a one year's leave. Dr. Ono is spending this time in working on high temperature emissivity of transparent materials in conjunction with Dr. DeWitt. Dr. Ono has received no AFOSR funds for his endeavors.
- (b) Dr. R. E. Taylor was asked by the Commission on Standardization of Methods for Thermophysical Properties Measurements to prepare an extended paper on direct heating methods. This chapter includes the high temperature emissometer developed as part of the AFOSR Grant.

